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(5*S**,6*R**)-1,7-Dioxadispiro[4.0.4.4]-tetradecane-2,8-dione

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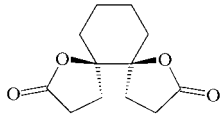
The title compound, $C_{12}H_{16}O_4$, (I), was prepared by oxidation of (5*S**,6*R**)-1,7-dioxadispiro[4.0.4.4]tetradecane-2,8-diol using silver(I) carbonate and posesses a *cis* configuration of the two five-membered-ring lactones fused spiro to the six-membered carbocycle, which has a chair conformation. It represents an exceptional structure for bis-tetrahydrofuran units, which are interesting building blocks in natural products. The synthesis, spectroscopic data and X-ray structural analysis are described. The crystal contains discrete molecules separated by normal van der Waals distances.



(I)

Experimental

Following the procedure of Nozaki *et al.* (1997) (5*S**,6*R**)-1,7-dioxadispiro[4.0.4.4]tetradecane-2,8-diol (300 mg, 1.31 mmol) and silver(I) carbonate (1.65 g, 5.98 mmol) in dry benzene (15 ml) were heated under reflux for 30 h. After cooling to room temperature, 2.5 g of celite was added and the mixture was stirred for an additional 10 min. The solid was filtered off and washed with methyl *tert*-butyl ether. After evaporation of the solvent, the crude product (light-yellow crystals, 260 mg) was recrystallized from 20 ml diethyl ether to give 212 mg (0.95 mmol, 72%) (5*S**,6*R**)-1,7-dioxadispiro[4.0.4.4]-tetradecane-2,8-dione as colourless crystals. Analysis calculated for $C_{12}H_{16}O_4$ (224.25 g mol^{-1}): C 64.27, H 7.19%; found: C 64.2, H 7.2%; MS (EI, 70 eV): *m*/*z* (%) = 224 (M^+, 16), 206 (15), 178 (12), 162 (5), 150 (4), 124 (100), 111 (71), 96 (27), 83 (19), 67 (13), 55 (45); IR (KBr) $\tilde{\nu}$ [cm^{-1}] = 2954 (*s*), 2940 (*s*), 2881 (*m*), 2863 (*s*), 1770 (*vs*), 1448 (*m*), 1280 (*s*), 1251 (*s*), 1193 (*s*), 1128 (*s*), 1035 (*s*), 983 (*s*), 925 (*s*); ^{1}H NMR (400 MHz, DMSO-d_6): δ (p.p.m.) = 1.46–1.76 (*m*, 6H), 1.87–1.95 (*m*, 2H), 2.00–2.18 (*m*, 4H), 2.50–2.61 (*m*, 2H), 2.71–2.81 (*m*, 2H); ^{13}C NMR (100 MHz, DMSO-d_6): δ (p.p.m.) = 20.96 (CH_2), 26.43 (CH_2), 28.06 (CH_2), 33.00 (CH_2), 87.80 (Cq), 176.02 (Cq, C═O); m.p.: 394 K.

Crystal data

$C_{12}H_{16}O_4$	D_x = 1.351 Mg m^{-3}
M_r = 224.25	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 9511 reflections
a = 6.8821 (2) Å	θ = 3.37–27.47°
b = 13.4754 (4) Å	μ = 0.101 mm^{-1}
c = 12.0807 (4) Å	T = 291 (1) K
β = 100.3383 (19)°	Block, colourless
V = 1102.16 (6) Å^3	0.40 × 0.39 × 0.26 mm
Z = 4	

Data collection

Nonius KappaCCD diffractometer	1552 reflections with $I > 2\sigma(I)$
Method: 281 frames *via* ω-rotation ($\Delta\omega$ = 1°) and 2 × 20 s per frame with three sets at different κ angles	R_{int} = 0.031
	θ_{max} = 27.47°
	$h = -8 \rightarrow 8$
	$k = -17 \rightarrow 17$
9511 measured reflections	$l = -15 \rightarrow 15$
2492 independent reflections	Intensity decay: none

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0627P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.038	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2)$ = 0.101	$\Delta\rho_{max}$ = 0.157 e Å^{-3}
S = 0.953	$\Delta\rho_{min}$ = −0.148 e Å^{-3}
2492 reflections	Extinction correction: *SHELXL*97
210 parameters	Extinction coefficient: 0.035 (6)
All H-atom parameters refined	

All H atoms were located in a Δ map and refined isotropically [C—H 0.917 (15)–1.023 (15) Å].

Data collection: *COLLECT Software* (Nonius, 1998); cell refinement: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1996); data reduction: *DENZO* and *SCALEPACK*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); software used to prepare material for publication: *SHELXL*97 and *PARST*95 (Nardelli, 1995).

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